

## Fabrication of metal-containing phenolic resins and control of their pore structures

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### Introduction

Phenolic resin (RF gel) prepared by the reaction of resorcinol and formaldehyde is useful as a porous carbon precursor because it can be synthesized in a water-soluble system, has a high carbonization yield, and shrinks little upon heat treatment. Furthermore, when hard carbon using phenol resin as a precursor is used as a negative electrode for Na-ion secondary batteries, a reversible capacity exceeding 300 mAh<sup>-1</sup> can be obtained [1]. Recently, it has been reported that the reversible capacity is improved in hard carbon electrodes obtained from precursors containing metallic elements [2].

In this study, metal-containing phenolic resins with a co-continuous structure were prepared by a sol-gel method involving phase separation with the aim of developing high-capacity hard carbon electrodes.

### Experimental Procedures

Resorcinol and metal chlorides (ZnCl<sub>2</sub>, MnCl<sub>2</sub>, MgCl<sub>2</sub>) were dissolved in a water/ethanol mixture. Formaldehyde was then added to the resulting solution and stirred at room temperature for 1 minute to obtain a homogeneous solution. The resulting solution was allowed to gel and age for 48 hours at 60 °C, and then dried at 60 °C to obtain dried gel. Metal-containing porous carbon was then prepared by calcination of the dried gel at 800 -1400 °C. Metal-free porous carbon was also prepared in the same way by adding 0.01 M hydrochloric acid as an acid catalyst for comparison. The pore structure of the samples was evaluated by scanning electron microscopy (SEM) observation and energy dispersive X-ray spectroscopy (EDX).

### Results and Discussion

Fig. 1 shows SEM images of the precursor gels obtained. For all precursor gels, the phase separation tendency decreased with increasing ethanol content. By optimizing the amount of ethanol added, porous gels with a three-dimensionally continuous co-continuous structure of the gel backbone and macropores, respectively, were obtained. Fig. 2 shows the EDX elemental mapping of the precursor gels. It is shown that the metal elements Zn, Mn, and Mg are homogeneously distributed in the precursor gels, respectively.

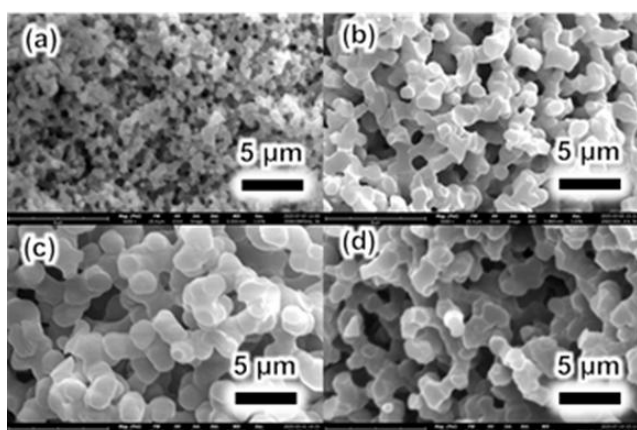


Fig. 1 SEM images of the as-dried RF gels: (a) without metal (b) Zn-containing, (c) Mn-containing and (d) Mg-containing RF gels.

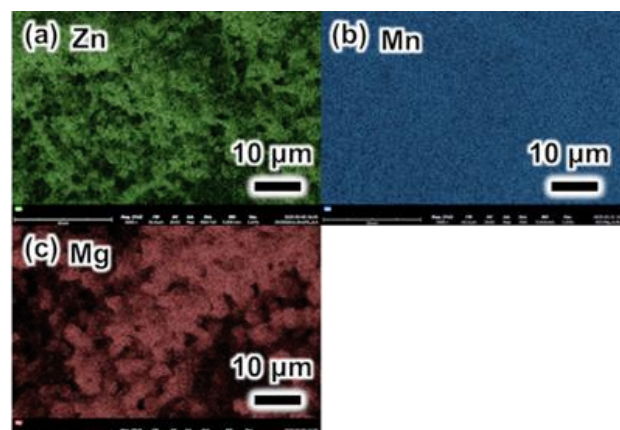


Fig. 2 EDX mapping images of (a) the as-dried Zn-doped RF gel, (b) the as-dried Mn-doped RF gel and (c) as-dried Mg-doped RF gel.

### References

- [1] G. Hasegawa et al., *ChemElectroChem* **2**, 1917(2015).
- [2] A. Kamiyama et al., *Angew. Chem. Int. Ed.* **60**, 5114(2021).